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(21) International Application Number: PCT/GB94/01342 (22) International Filing Date: 21 June 1994 (21.06.94) (30) Priority Data: 9313128.2 24 June 1993 (24.06.93) GB (71) Applicant (for all designated States except US): COUR- TAULDS FIBRES (HOLDINGS) LIMITED [GB/GB]; 50 George Street, London W1A 2BB (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): PEARSON, Leslie [GB/GB]; 30 Church Mews, Spondon, Derby DE21 7NQ (GB). TAYLOR, James, Martin [GB/GB]; 146 Field Lane, Alveston, Derby DE2 6GW (GB). (74) Agent: NEWBY, John, Ross; J.Y. & G.W. Johnson, Furnival House, 14-18 High Holborn, London WC1V 6DE (GB).		(81) Designated States: AM, AT, AT (Utility model), AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DE (Utility model), DK, ES, FI, GB, HU, JP, KE, KG, KP, KR, KZ, LK, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, US, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: FABRIC TREATMENT (57) Abstract The degree of fibrillation and/or the tendency to fibrillation of lyocell fabric can be reduced by treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin, heating the treated fabric to cure the resin, and washing and drying the fabric. The treatment can be applied to dyed fabric.		

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FABRIC TREATMENT**Background of the Invention****1. Field of the Invention**

This invention is concerned with methods of reducing the
5 degree of fibrillation and fibrillation tendency of fabric
made from solvent-spun cellulose fibre, also known as lyocell
fibre.

2. Description of Related Art

It is known that cellulose fibre can be made by extrusion
10 of a solution of cellulose in a suitable solvent into a
coagulating bath. This process of extrusion and coagulation
is referred to as "solvent-spinning", and the cellulose fibre
produced thereby is referred to as "solvent-spun" cellulose
fibre. One example of such a process is described in US-
15 A-4,246,221, the contents of which are incorporated herein by
way of reference. Cellulose is dissolved in a solvent such as
a tertiary amine N-oxide, for example N-methylmorpholine N-
oxide. The resulting solution is extruded through a suitable
die to produce filaments, which are coagulated, washed in
20 water to remove the solvent, and dried. The filaments are
generally cut into short lengths at some stage after
coagulation to form staple fibre. It is also known that
cellulose fibre can be made by extrusion of a solution of a
cellulose derivative into a regenerating and coagulating bath.
25 One example of such a process is the viscose process, in which
the cellulose derivative is cellulose xanthate. Both such
types of process are examples of wet-spinning processes.
Solvent-spinning has a number of advantages over other known
processes for the manufacture of cellulose fibre such as the
30 viscose process, for example reduced environmental emissions.

As used herein, the term "lyocell fibre" means a
cellulose fibre obtained by an organic solvent-spinning
process, in which the organic solvent essentially comprises
a mixture of organic chemicals and water, and in which
35 solvent-spinning involves dissolving cellulose and spinning

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without formation of a derivative of the cellulose. As used herein, the terms "solvent-spun cellulose fibre" and "lyocell fibre" are synonymous. As used herein, the term "lyocell fabric" means a fabric woven or knitted from a plurality of 5 yarns, at least some of which yarns contain lyocell fibre, alone or in blend with other type(s) of fibre.

Fibre may exhibit a tendency to fibrillate, particularly when subjected to mechanical stress in the wet state. Fibrillation occurs when fibre structure breaks down in the 10 longitudinal direction so that fine fibrils become partially detached from the fibre, giving a hairy appearance to the fibre and to fabric containing it, for example woven or knitted fabric. Dyed fabric containing fibrillated fibre tends to have a "frosted" appearance, which may be 15 aesthetically undesirable. Such fibrillation is believed to be caused by mechanical abrasion of the fibres during treatment in a wet and swollen state. Wet treatment processes such as scouring, bleaching, dyeing and washing inevitably subject fibres to mechanical abrasion. Higher temperatures 20 and longer times of treatment generally tend to produce greater degrees of fibrillation. Lyocell fabric appears to be particularly sensitive to such abrasion and is consequently often found to be more susceptible to fibrillation than fabric made from other types of cellulose fibre. In particular, 25 cotton fabrics have an inherently very low fibrillation tendency.

EP-A-538,977 discloses that fibrils can be removed from fibrillated woven lyocell fabric by treatment with a solution of a cellulase. Cellulases are enzymes which catalyse the 30 hydrolysis of cellulose. However, such treatment is not as effective as could be desired, and disposal of used solutions of the enzyme may pose environmental problems.

It has been known for many years to treat cellulose fabric with a crosslinking agent to improve its crease 35 resistance, as described for example in Kirk-Othmer's

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Encyclopaedia of Chemical Technology, third edition, Volume 22 (1983), Wiley-Interscience, in an article entitled "Textiles (Finishing)" at pages 769-790, and in an article by H. Petersen in Rev. Prog. Coloration, Vol 17 (1987), pages 7-22. Crosslinking agents may sometimes be referred to under other names, for example crosslinking resins, chemical finishing agents and resin finishing agents. Crosslinking agents are small molecules containing a plurality of functional groups capable of reacting with the hydroxyl groups in cellulose to form crosslinks. In the conventional type of finishing process, a cellulosic fabric is first treated with a crosslinking agent, for example by application from a pad bath, dried, and then heated to cure the resin and induce crosslinking (pad-dry-cure). It is known that crease-resistant finishing treatments embrittle cellulose fabric with consequent loss of abrasion resistance, tensile strength and tear strength.

The first crosslinking systems were based on formaldehyde, urea-formaldehyde and melamine-formaldehyde resins. These suffered a number of problems. The treatment caused temporary stiffening of the fabric because of the presence of externally adhering resin. Treated fabric was liable to liberate objectionable odours on storage. These odorous substances included the amine catalysts used to cure the resin and the toxic chemical formaldehyde. It was therefore considered necessary to wash the treated fabric to remove externally adhering resin and byproducts of resin formation capable of giving rise to objectionable odours. Such washing and subsequent drying of the treated fabric added to the cost of the process.

Such systems have largely been replaced by systems containing the so-called "low-formaldehyde resins" and "zero-formaldehyde resins" as crosslinking agents. One known class of such agents consists of the N-methylol resins, that is to say small molecules containing two or more N-hydroxymethyl or N-alkoxymethyl, in particular N-methoxymethyl, groups. N-

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methylool resins are generally used in conjunction with acid catalysts chosen to improve crosslinking performance. In a typical process, a solution containing about 5-9% by weight N-methylool resin crosslinking agent and 0.4-3.5% by weight
5 acid catalyst is padded onto dry cellulosic fabric to give 60-100% by weight wet pickup, after which the wetted fabric is dried and heated to cure and fix the crosslinking agent. Fabrics treated with low-formaldehyde or zero-formaldehyde resins generally do not exhibit temporary stiffening and do
10 not release objectionable odours. Cured flat fabrics and finished garments are rarely washed prior to sale to the consumer.

Summary of the Invention

According to a first aspect of the invention, a method
15 for reducing the fibrillation tendency of lyocell fabric includes the steps of:

- a) treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin;
- b) heating the fabric under conditions effective to
20 cause reaction between the resin and the cellulose;
- c) washing the fabric; and
- d) drying the fabric.

In this and the other aspects of the invention, the fabric is treated with the crosslinking resin and heated to
25 cause reaction between the resin and the cellulose in the form of flat fabric. In one embodiment of this and the other aspects of the invention, which may be preferred, the fabric is washed and dried in the form of flat fabric and is thereafter suitable for cutting into pieces for the
30 manufacture of garments or other textile articles. In another embodiment of this and other aspects of the invention, the fabric is first made up into garments or other textile

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articles which are then washed and dried to complete the method of the invention.

According to a second aspect of the invention, a method for reducing the degree of fibrillation of lyocell fabric 5 includes the steps of:

- a) treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin;
- b) heating the fabric under conditions effective to cause reaction between the resin and the cellulose;
- 10 c) washing the fabric; and
- d) drying the fabric.

According to a third aspect of the invention, a method for providing a lyocell fabric which exhibits a low degree of fibrillation and has a low fibrillation tendency includes the 15 steps of:

- a) scouring and dyeing the fabric, thereby inducing fibrillation in the fabric;
- b) treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin;
- 20 c) heating the fabric under conditions effective to cause reaction between the resin and the cellulose;
- d) washing the fabric; and
- e) drying the fabric.

In this third aspect of the invention, the fabric may 25 optionally be bleached between the scouring and dyeing processes in step (a) and may optionally be dried between

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steps (a) and (b). After step (c), the fabric may exhibit such a high degree of fibrillation that textile articles made from it would be commercially unacceptable. After step (e), the fabric in the form of flat fabric or of textile articles
5 exhibits a very low and commercially desirable degree of fibrillation.

One class of preferred crosslinking resins consists of the N-methylol resins. Examples of suitable N-methylol resins are those described in the abovementioned articles in Kirk-
10 Othmer and by Petersen. Examples of such resins include 1,3-dimethylolethyleneurea (DMEU), 1,3-dimethylolpropyleneurea (DMPU) and 4,5-dihydroxy-1,3-dimethylolethyleneurea (DHDMEU). Other examples include compounds based on urones, triazinones and carbamates. Another example of a preferred class of
15 crosslinking agents consists of compounds based on 1,3-dialkyl-4,5-dihydroxy(alkoxy)ethyleneurea and its derivatives. A further example of a suitable crosslinking agent is melamine. Yet another example of a suitable crosslinking agent is butanetetracarboxylic acid (BTCA). More than one
20 type of crosslinking resin may be used.

Crosslinking agents for crease-resistant finishing of cellulose fabric are generally used in conjunction with a catalyst. The catalyst serves to accelerate the crosslinking reaction and curing and fixation of the resin. The method of
25 the invention preferably utilises such a catalyst when recommended for use with the chosen crosslinking agent. For example, N-methylol resins are preferably used in conjunction with an acid catalyst, for example an organic acid such as acetic acid or a mineral acid such as zinc nitrate or
30 magnesium chloride. Latent acids such as ammonium salts, amine salts and metal salts may be used. Mixed catalyst systems may be used.

The crosslinking agent and any catalyst are preferably applied to the fabric from solution, preferably in water. The
35 solution may be applied to the fabric in known types of ways,

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for example the solution may be padded on to the fabric or the fabric may be passed through a treatment bath of the solution. The fabric may be a woven or knitted fabric. The solution may contain at least about 2%, preferably about 3 to about 6%, by weight crosslinking agent. When a catalyst is used, the solution may contain at least about 1%, preferably about 1 to about 2%, by weight catalyst.

After treatment with crosslinking resin according to the invention, the fabric is heated to fix and cure the crosslinking agent. The fabric may also be dried. The heating step may precede, be part of, or follow the drying step. The time and temperature required in the heating step depend on the nature of the crosslinking agent and optional catalyst employed. After heating and optionally drying, the fabric may contain at least about 0.5%, preferably at least about 1.0%, more preferably at least about 2.0%, by weight of fixed crosslinking agent calculated on the weight of the cellulose. The fabric generally contains no more than about 4% by weight of fixed crosslinking agent calculated on the weight of the cellulose. It has generally been found that about 70 to 90% of the crosslinking agent in the wet fabric may become fixed to the cellulose.

The concentration of the resin in the bath is chosen according to the activity and curing efficiency of that resin, to give the desired value for resin fixed on fabric.

After heating and fixation, the fabric is washed and dried according to conventional procedures for cellulose fabrics.

Fabric treated according to the invention exhibits a very low degree of fibrillation. This is particularly

surprising in that treatment with crosslinking agents is generally believed to reduce the abrasion resistance of cellulosic fabrics. The method of the invention requires an

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additional wet processing step, and as mentioned hereinabove, such wet processing steps are known to cause fibrillation of lyocell fabric. Fabric treated according to the invention has excellent resistance to fibrillation compared with untreated fabric. Fabric treated according to the invention is suitable for the manufacture of textile articles such as garments. Such textile articles may be laundered with only little or slow loss of the reduction in fibrillation tendency.

The method of the invention is applicable to fabric which has already been dyed, including fabric dyed by processes such as rope-dyeing which are known to cause mechanical abrasion. This is an advantage of the invention, because it is known that rope-processing of a fabric generally improves bulking and relaxation of the fabric, leading to superior handle. The method of the invention can be used on fabric which is already fibrillated, even severely fibrillated. It has surprisingly been found that when fabric exhibiting a high degree of fibrillation is treated according to the method of the invention, the treated fabric generally exhibits a very low level of fibrillation. For most applications, fabric which exhibits a high degree of fibrillation is considered to be of substandard quality, with the consequence that additional expensive processing steps are not considered to be justified. It is a particular advantage of the invention that it permits substandard fabric to be converted into first quality fabric and textile articles.

Materials were assessed for degree of fibrillation using the method described below as Test Method 1.

Test Method 1 (Assessment of Fibrillation)

There is no universally accepted standard for assessment of fibrillation, and the following method was used to assess Fibrillation Index (F.I.). Samples of fibre were arranged into a series showing increasing degrees of fibrillation. A standard length of fibre from each sample was then measured

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and the number of fibrils (fine hairy spurs extending from the main body of the fibre) along the standard length was counted. The length of each fibril was measured, and an arbitrary number, being the product of the number of fibrils multiplied 5 by the average length of each fibril, was determined for each fibre. The fibre exhibiting the highest value of this product was identified as being the most fibrillated fibre and was assigned an arbitrary Fibrillation Index of 10. A wholly unfibrillated fibre was assigned a Fibrillation Index of zero, 10 and the remaining fibres were evenly ranged from 0 to 10 based on the microscopically measured arbitrary numbers.

The measured fibres were then used to form a standard graded scale. To determine the Fibrillation Index for any other sample of fibre, five or ten fibres were visually 15 compared under the microscope with the standard graded fibres. The visually determined numbers for each fibre were then averaged to give a Fibrillation Index for the sample under test. It will be appreciated that visual determination and averaging is many times quicker than measurement, and it has 20 been found that skilled fibre technologists are consistent in their rating of fibres.

Fibrillation Index of fabrics can be assessed on fibres drawn from the surface of the fabric. Woven and knitted fabrics having F.I. of more than about 2.0 to 2.5 exhibit an 25 unsightly appearance.

Description of Preferred Embodiments

The invention is illustrated by the following Examples. In each case, the never-dried fibre used was prepared by extruding a solution of cellulose in N-methylmorpholine N- 30 oxide (NMMO) into an aqueous bath and washing the fibre so formed with water until it was essentially free of NMMO.

Examples

A 100% lyocell spun yarn woven fabric exhibiting zero

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F.I. was desized, scoured and dyed in a jet dyeing machine. Desizing was carried out using a 1.5 g/l aqueous solution of a commercially-available amylase preparation at pH 6.5-7.5 for 45 minutes at 70°C. Scouring was carried out using an aqueous solution containing 2 g/l sodium carbonate and 2 g/l anionic detergent for 60 minutes at 95°C. Dyeing was carried out using an aqueous solution containing 4% by weight on fabric of the dyestuff Procion Navy HE-R 150 (Procion is a Trade Mark of Zeneca plc), 80 g/l sodium sulphate and 20 g/l sodium carbonate for 120 minutes at 85°C. The fabric was rinsed with water, first at 70°C and then at ambient temperature; soaped off using an aqueous solution containing 2 g/l Sandopur SR (Sandopur is a Trade Mark of Sandoz AG) for 20 minutes at 95°C; hydroextracted; and dried. This procedure is a typical rope treatment process for cellulosic fabrics. The treated fabric was severely fibrillated, and exhibited F.I. 4.5.

Samples of dyed fabric were padded with aqueous solutions containing varying amounts of the low-formaldehyde resin DHDMEU (supplied under the Trade Mark Arkofix NG conc by Hoechst AG). The solutions contained an acid-liberating catalyst as recommended by the resin supplier at 25% by weight on weight of Arkofix NG conc. The samples of fabric were then dried at 110°C and the resin flash cured for 30 seconds at 180°C. They were then reloaded on the jet dyeing machine and scoured twice as before. The samples were then hydroextracted, padded wet-on-wet with an aqueous dispersion containing 50 g/l of a silicone-based softener (available under the Trade Mark Rucofin A0736 from Rudolf Chemicals Ltd.), and dried at 110°C. The results were as shown in Table 1:

30

Table 1

Resin in bath % w/w	0.0	1.0	2.0	4.0	6.0	8.0
Resin fixed on fabric % w/w	0.0	0.5	1.0	2.0	3.0	4.0
F.I.	5.8	1.9	1.5	0.5	0.4	0.3

The figures for the amount of resin fixed on fabric are

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estimated figures, based on 70% active solids in Arkofix NG conc, 80% expressed liquor, and 85% curing efficiency.

It can be seen that the F.I. of the untreated fabric increased from 4.5 as expected during these additional wet processing steps, whereas in contrast treatment with resin actually reduced the F.I. of the fabric from 4.5 to a commercially acceptable level of 1.9 or less.

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CLAIMS

1. A method for providing a lyocell fabric which exhibits a low degree of fibrillation and has a low fibrillation tendency, characterised in that it includes the steps of:

- 5 a) scouring and dyeing the fabric, thereby inducing fibrillation in the fabric;
- b) treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin;
- 10 c) heating the fabric under conditions effective to cause reaction between the resin and the cellulose;
- d) washing the fabric; and
- e) drying the fabric.

2. A method according to claim 1, characterised in that the dyeing step is carried out on fabric in rope form.

- 15 3. A method according to claim 1 or claim 2, characterised in that it includes the step of bleaching the fabric subsequent to the scouring step and prior to the dyeing step.

20 4. A method according to any one of claims 1 to 3, characterised in that it includes the step of drying the fabric subsequent to the dyeing step and prior to the step of treating the fabric with the crosslinking resin.

25 of: 5. A method for reducing the fibrillation tendency of lyocell fabric, characterised in that it includes the steps of:

- a) treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin;

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- b) heating the fabric under conditions effective to cause reaction between the resin and the cellulose;
 - c) washing the fabric; and
 - d) drying the fabric.
- 5 6. A method for reducing the degree of fibrillation of lyocell fabric, characterised in that it includes the steps of:
- a) treating the fabric with a low-formaldehyde or zero-formaldehyde crosslinking resin;
 - 10 b) heating the fabric under conditions effective to cause reaction between the resin and the cellulose;
 - c) washing the fabric; and
 - d) drying the fabric.
- 15 7. A method according to any preceding claim, characterised in that the washing and drying steps which follow the heating step are carried out on fabric in flat form.
- 20 8. A method according to any of claims 1 to 6, characterised in that the fabric is made up into garments or other textile articles after the heating step and prior to the washing step.
- 25 9. A method according to any preceding claim, characterised in that the crosslinking resin comprises at least one N-methylol resin.
- 25 10. A method according to claim 9, characterised in that the N-methylol resin is used in conjunction with an acid catalyst.

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11. A method according to any one of claims 1 to 8, characterised in that the crosslinking resin comprises at least one resin selected from the group consisting of urones, triazinones, carbamates, 1,3-dialkyl-4,5-dihydroxy(alkoxy)-5 ethyleneurea and derivatives thereof, melamine and butanetetracarboxylic acid.

12. A method according to any preceding claim, characterised in that the treatment step is carried out by applying an aqueous solution containing 3 to 6 percent by 10 weight crosslinking agent to the fabric.

13. A method according to any preceding claim, characterised in that, after the heating step, the fabric contains at least about 2 percent by weight fixed crosslinking agent based on the weight of cellulose.

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 5 D06M15/423 D06M13/432 D06M13/425 D06M13/192

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 D06M D01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	EP,A,0 538 977 (COURTAULDS PLC) 28 April 1993 cited in the application see page 5, line 7 - line 14; claims ---	1
P,A	WO,A,94 09191 (COURTAULDS FIBRES LTD.) 28 April 1994 see the whole document ---	1-13
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 013, no. 575 (C-667) 19 December 1989 & JP,A,01 239 167 (ASAHI CHEM. IND. CO. LTD.) 25 September 1989 see abstract ---	1
A	US,A,4 277 243 (ROWLAND) 7 July 1981 see claims ---	1-13
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A	TEXTILE RESEARCH JOURNAL., vol.62, no.6, June 1992, US pages 349 - 356, XP274707 NOELIE R. BERTONIERE AND WALTER D. KING 'Pore Structure of Cotton Fabrics Crosslinked with Formaldehyde-Free Reagents' see page 352, left column, paragraph 2 -paragraph 3 ---	1,11
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